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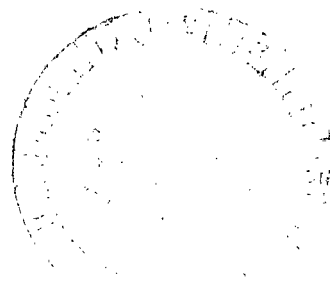
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National Aeronautics
and Space Administration

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1978

PRESSURELESS SINTERED SIALONS WITH LOW AMOUNTS OF SINTERING AID

by Alan Arias

Lewis Research Center

SUMMARY

The present investigation was undertaken to produce single-phase β' -Sialons (silicon-aluminum-oxygen-nitrogen ceramics) by pressureless sintering mixtures of separately milled β -silicon nitride (β - Si_3N_4), aluminum nitride (AlN), and silica (SiO_2) with small amounts of yttria (Y_2O_3) as a sintering aid. Two Sialons of compositions $\text{Si}_{2.6}\text{Al}_{0.393}\text{Y}_{0.007}\text{O}_{0.4}\text{N}_{3.6}$ (Sialon A) and $\text{Si}_{2.6}\text{Al}_{0.386}\text{Y}_{0.014}\text{O}_{0.4}\text{N}_{3.6}$ (Sialon B) and densities of 3.13 and 3.17 grams per cubic centimeter, respectively, were made by pressureless sintering cold-pressed powder compacts at 1760°C for 4 hours in stagnant nitrogen. This indicated that very small amounts of additives (or impurities) are required to bring about pressureless sintering of very fine powders compounded into Sialons. Sialons A and B had four-point moduli of rupture (MOR) ranging from about 460 megapascals at room temperature to 155 megapascals at 1400°C . The MOR of Sialon B was generally higher than that of Sialon A.

The oxidation resistance of Sialon A at 1400°C in air is slightly better than that of Sialon B. A comparison with data from other studies shows that Sialons A and B are more oxidation resistant than either hot-pressed Si_3N_4 or a similar Sialon with 2.5 wt % Y_2O_3 , but less so than a Sialon with no additives but with much more aluminum. In general, Sialons with well-defined compositions and having reportedly higher strengths have lower oxidation resistance than Sialons A and B; conversely, Sialons with higher oxidation resistance have lower strength than Sialons A and B. From these data we may surmise that increasing Y_2O_3 additions will increase the strength of Sialons but decrease their oxidation resistance.

Pressureless sintering Sialons A and B reduced their carbon content to negligible levels and reduced their oxygen content proportionally, presumably because of the reaction of carbon with SiO_2 to form silicon and carbon monoxides.

INTRODUCTION

The demand for ever higher operating temperatures in heat engines has created the need for materials with high oxidation resistance and strength. Silicon carbide- and silicon nitride-base ceramics have properties that make them likely candidates for applications in advanced heat engines, and among the new types of silicon nitride-base ceramics that have received a great deal of attention since they were first reported (refs. 1 and 2) are the Sialons, so-called because they are made up of silicon (Si), aluminum (Al), oxygen (O), and nitrogen (N) atoms. The attention given these Sialon ceramics stems mainly from the reportedly low thermal expansion, high oxidation resistance, and ease of fabrication by conventional, pressureless sintering techniques. In addition, the single-phase β' -Sialons (refs. 3 and 4) vary greatly in composition and, consequently, could well vary correspondingly in physical properties. This variation in properties might then allow us to tailor the composition to the intended application.

The main objectives of the present investigation were to produce β -Sialons with high silicon nitride (Si_3N_4) content and low amounts of sintering aid by pressureless sintering methods and to determine the strength and oxidation resistance of the resulting ceramics. Additional objectives were to produce these Sialons from β - Si_3N_4 and to use a novel milling approach in their preparation.

To carry out these objectives β - Si_3N_4 , aluminum nitride (AlN), and silica (SiO_2) were ball milled separately. The resulting powders were chemically analyzed and by using these analyses as a guide, the milled powders and small amounts of yttria (Y_2O_3), as a sintering aid, were compounded into the desired compositions. Cold pressed bars of these compositions were sintered in nitrogen. The sintered bars were then used to determine the oxidation resistance and the modulus of rupture as a function of temperature.

MATERIALS

The materials used in this investigation were powdered β - Si_3N_4 , AlN, SiO_2 , and Y_2O_3 . These materials are characterized in table I together with other materials used in processing them.

EQUIPMENT

Two types of ball mills were used in this investigation: nickel-lined ball mills of 1500 cubic centimeter capacity with ~3700 grams of nickel shot (0.63 to 1.27 cm diam); and alumina ball mills of 1400 cubic centimeter capacity with ~1200 grams of alumina balls (1.27 cm diam). The rest of the equipment used in this investigation (presses,

cold pressing dies, furnaces, tensile testers, analytical equipment, etc.) was standard laboratory equipment.

PROCEDURES

The procedures used for the preparation and testing of Sialon specimens are outlined in the flow diagram of figure 1. These procedures will be briefly described here.

Milling. - Batches of 200-gram, -325-mesh, β - Si_3N_4 were milled for 300 hours in the nickel-lined ball mills with 500 milliliters of distilled water as the milling fluid. The -325-mesh, SiO_2 was also milled in nickel-lined mills. The 150-gram powder batches were milled 130 hours with 500 milliliters of distilled water as the milling fluid. The 300-gram batches of -325-mesh AlN were milled 100 hours in alumina mills with 500 milliliters of n-heptane as the milling fluid.

Removal of pickup. - During ball milling, materials resulting from the wear of the balls and mills (pickup) contaminate the milled powders. In most cases this pickup is detrimental to the properties of the Sialons and must be removed. After separating the slurries of milled β - Si_3N_4 and SiO_2 from the nickel shot by sieving, most of the nickel pickup in the milled powders was removed magnetically. The slurries were heated, stirred, treated with 100 grams of concentrated nitric acid and then centrifuged to remove most of the liquid. To wash the powders, distilled water was added to the moist powder and the mixture was stirred while being heated. The water was then removed by centrifuging. The washing-centrifuging procedure was repeated twice more. The resulting powder cakes were vacuum dried at about 100°C and pulverized in a Waring blender. The powders were kept in air-tight containers until ready for use.

No attempt was made to remove the alumina pickup (~ 1 wt %) from the AlN milled powders, since Sialons can be compounded with alumina. The slurries resulting from milling AlN were dried in a stream of dry nitrogen and then processed the same as the β - Si_3N_4 and SiO_2 milled powders.

The Y_2O_3 powder was not ball milled because the as-received powder was already quite fine.

Chemical and BET analyses. - The milled and the as-received powders were analyzed for oxygen, for carbon, and (spectrographically) for trace elements. The chemical analyses of the milled powders and Y_2O_3 were required to calculate the amounts of each of these powders necessary to compound the desired Sialon. In these calculations it was assumed that all the carbon in the powders would react with SiO_2 to form SiO and CO gases (ref. 5).

The specific surface areas of the powders were determined by the BET (Brunauer, Emmett, and Teller) method.

Mixing. - The calculated amounts of milled β - Si_3N_4 , milled AlN , milled SiO_2 , and as received Y_2O_3 were weighed in an analytical balance and placed in a polyethylene bottle with about twice their weight in stainless-steel balls, 70 wt % ethanol, and 5 wt % silicone oil (as a temporary binder). The powders were mixed for 1 hour at 100 rpm. The resulting slurry was dried with constant stirring at about 100°C . After removing the balls by sieving, the resulting powder agglomerates were broken up in a Waring blender.

Cold pressing. - The powder mixtures were shaped into bars approximately 3.81 by 0.92 by 0.47 centimeters in a double-acting steel die at a pressure of 207 megapascals (30 ksi). These bars were then encased in plastic bags, evacuated, sealed, and isostatically cold pressed at 483 megapascals (70 ksi).

Removing the fugitive binder. - The silicone oil binder was removed from the bars by heating them in flowing nitrogen at 450°C for about 1 hour.

Sintering. - The bars were placed in a tungsten boat and sintered in a furnace with graphite heating elements for 4 hours at $1760 \pm 20^\circ\text{C}$ in stagnant nitrogen at 34.5 kilopascals (5 psi) gage pressure. The temperature was controlled and monitored with W/W-26Re thermocouples. The sintering specimens were surface ground into 0.635-by-0.318-by-~2.54-centimeter test bars, and the edges beveled 0.12 millimeter. The bars used for oxidation tests were further polished to a mirror finish on a 10-micrometer diamond lap; the final size was about 0.630 by 0.315 by 1.5 centimeters.

Testing. - The ground bars were used to determine the four-point MOR from room temperature to 1400°C in air. The SiC MOR fixture used had a bottom span of 1.905 centimeters (3/4 in.) and a top span of 0.953 centimeter (3/8 in.). A SiC muffle furnace mounted on an Instron tensile tester was used to heat up the bars and the fixture to the test temperature. The bars were bend tested at a crosshead speed of 0.051 centimeter per minute (0.020 in./min).

Oxidation tests were carried out in air at 1400°C . During the tests the bars were held in a platinum crucible having two side holes and a loosely fitting cover, so as to allow access of air to the Sialon bars. At various times the bars were cooled and weighed in an analytical balance with microgram sensitivity. Weight gains as a function of time curves were determined from the weighing data.

The densities of the sintered Sialons were determined by the water immersion method. Chemical, X-ray, scanning electron microscopy (SEM), microprobe, and optical microscopy analyses of the sintered and ground Sialons were done.

Auxiliary experiment. - To compare densities, two Sialons of the same compositions as Sialons A and B were hot-pressed in a graphite die at $1750 \pm 25^\circ\text{C}$ and 27.6 megapascals (4000 psi) for 1 hour in flowing nitrogen. The resultant Sialons were assumed to be 100 percent dense.

RESULTS AND DISCUSSION

Sialons Investigated

The two Sialon compositions used in this investigation were compounded from finely milled Si_3N_4 , AlN , and SiO_2 powders (chemically characterized in table II) with small amounts of Y_2O_3 (table I) as a sintering aid. These two Sialons, which are designated A and B in table III, were compounded with 0.56 and 1.11 wt % Y_2O_3 , respectively. It was assumed for the calculations that the Y_2O_3 would go into solid solution during sintering, replacing an equivalent amount of Al_2O_3 moles in the Sialon structure. Based on this assumption, Sialons A and B fall within the β' homogeneity range defined by the equation $\text{Si}_{6-z}\text{Al}_z\text{O}_z\text{N}_{8-z}$ (refs. 3 and 4). However, in this work the equivalent equation $\text{Si}_{3-x}\text{Al}_x\text{O}_x\text{N}_{4-x}$ will be used to allow comparison with other Sialon compositions (e. g. , those of refs. 6 and 7). In this work $x = 0.4$. The reason for choosing compositions with $x = 0.4$ is that they have almost the most Si_3N_4 that it is possible to have without falling outside the β' field. This choice of x also limits the Y_2O_3 additions. The minimum Y_2O_3 additions are determined by their effect on the sinterability of the material. For example, the same Sialons with less than about 0.25 wt % Y_2O_3 cannot be pressureless sintered significantly; those without Y_2O_3 could not be consolidated to more than 2.64 grams per cubic centimeter even after hot pressing at 1750°C and 27.6 megapascal (4000 psi) for 1 hour in nitrogen. Sialons with 2.5 and higher wt % Y_2O_3 additions have been reported (ref. 6), and this allows us to compare the gross effects of Y_2O_3 on MOR and oxidation resistance.

Modulus of Rupture

Four-point MOR as a function of temperature plots of Sialons A and B are shown in figure 2. The number of bars tested and the range of MOR values are also indicated in this figure. Both Sialons have about the same MOR at room temperature (415 to 500 MPa) and at 1400°C (140 to 165 MPa). At intermediate temperatures Sialon A generally has a lower MOR than Sialon B. Both Sialons show an accelerated drop in strength with temperature beyond the 1000° to 1200°C range. This drop is common to most of the Sialons reported in the literature.

Also shown in figure 2 are the MOR versus temperature data for Sialons from references 6 to 8. At 1400°C the Sialons from references 7 and 8 have about the same MOR as Sialons A and B, and the Sialon from reference 6 (with 2.5 wt % Y_2O_3) has a higher MOR. At lower temperatures Sialons A and B have generally higher MOR's than the Sialons from references 7 and 8. Other Sialons with higher MOR's in the room temperature to 1400°C range have been reported (ref. 9); but these were three-point MOR's,

and their compositions were not defined. For these reasons, further comparisons were meaningless. The MOR's of Sialons A and B could probably be improved. The processing variables (sintering time and temperature, powders particle size, composition, etc.) have not been optimized, and there is considerable room for improvement in other properties as well as in MOR.

Oxidation

The weight increases with time on oxidizing Sialons A and B in air at 1400° C are plotted in figure 3. For comparison, the 1400° C oxidation behavior for hot-pressed Si_3N_4 (ref. 10) and for two Sialons from references 6 and 8 are also plotted. From the data in this figure it is calculated that Sialons A and B gain weight at a rate of about 0.004 mg/cm²-hr after being oxidized for 35 hours. Under similar conditions, the hot-pressed Si_3N_4 oxidizes at a rate of 0.013 mg/cm²-hr; the Sialon with 2.5 wt % Y_2O_3 oxidizes at ~0.043 mg/cm²-hr; and the TLP Sialon with the much higher Al content (known to increase oxidation resistance) oxidizes at 0.0011 mg/cm²-hr. Thus, the Sialons from the present investigation have better oxidation resistance than the hot-pressed Si_3N_4 (ref. 10) and the Sialons with 2.5 wt% Y_2O_3 (ref. 6). The fact that Sialons A and B have better oxidation resistance than the 2.5-wt% Y_2O_3 Sialon is assumed to be due to their lower Y_2O_3 content. In addition to the Sialons with 2.5 wt% Y_2O_3 , a 5-wt% Y_2O_3 Sialon (ref. 6) also has generally higher strength and (as already mentioned) much lower oxidation resistance than the Sialons A and B. Thus, it appears that increasing Y_2O_3 additions increases the strength of Sialons but decreases their oxidation resistance.

As also said above, the transient liquid phase (TLP) Sialon (ref. 8) has a higher oxidation resistance than Sialons A and B. However, aluminum increases the oxidation resistance of Sialons (ref. 11), and the TLP Sialon has a much higher aluminum content than Sialons A and B. Thus, Sialons with higher aluminum content made by the methods described herein should have better oxidation resistance.

X-ray diffraction analyses of the oxidized layers of Sialons A and B show α -cristobalite (strong), β - Si_3N_4 (medium) and mullite (weak). The β - Si_3N_4 may have been unoxidized Sialon scraped off the surface as the X-ray sample was taken.

Microstructures

In figure 4 is shown an optical photomicrograph of Sialon A. It shows small amounts of a light gray phase, smaller amounts of (dark looking) holes or pull outs, and very small amounts of a bright, metallic-looking phase in a fairly uniform matrix. The photomicrographs of Sialon B are similar.

Figure 5 is a photomicrograph of the room temperature fracture surface of Sialon A taken by scanning electron microscopy (SEM). The SEM photomicrographs of Sialons A and B are very similar, and both show equiaxed grains about 1.1-micrometer average diameter.

Electron microprobe analyses of Sialons A and B showed that the light gray spots seen in the optical photomicrographs have higher amounts of aluminum, iron, calcium, and magnesium and lower silicon and yttrium than the matrix. In addition, X-ray diffraction of these Sialons shows only the β' phase. From this it is surmised that the light gray spots are glass. Electron microprobe analysis showed that the bright, metallic-looking spots are richer in iron (and possibly in calcium) than the matrix. This bright phase may be an Fe(Ca)-Si alloy or intermetallic compound, but the amount of this phase is too small for identification by X-ray diffraction. Electron microprobe scans of the matrix of Sialons A and B showed that their compositions were essentially constant.

Chemical and Surface Area Analyses and Implications

Table I shows the chemical analyses of the as-received powders and table II of some of the same powders after milling. Table II also includes some milling and processing data. Comparison of tables I and II shows that, after milling and leaching, the nickel content of Si_3N_4 increased by about 500 ppm and that of the SiO_2 by about 900 ppm. From the weight loss of the balls and mill it was calculated that the AlN picked up 1.0 wt % alumina.

From the chemical analyses of the milled powders and Y_2O_3 only the elements silicon, aluminum, yttrium, oxygen, and carbon were taken into account for computing the amounts of powders required for compounding the Sialons. Spectrographically determined impurities other than silicon or aluminum were regarded as part of the major metallic elements (Al, Si, or Y) in the powder.

The specific surface areas of the Si_3N_4 was increased 6.75 times by milling. Those of the AlN and SiO_2 were increased by 4.5 and 29.5 times, respectively. The large specific surface area of the milled powders and Y_2O_3 would account for the relatively small amounts of sintering aid (Y_2O_3) required to effect pressureless sintering of Sialons A and B to over 98 percent of their theoretical density. It is noteworthy that the 0.56 wt % Y_2O_3 used in Sialon A is equivalent (in number of covalent bonds) to about 0.19 wt % MgO. This gives us an idea of the very small amounts of additive (or impurities) that can bring about densification of Sialons made from fine powders and of the importance of using high purity raw materials.

As already stated, excess SiO_2 was used for compounding Sialons A and B to allow for its loss by reaction with C to form CO and SiO gases during sintering. The analyses of sintered Sialons (table III) show very low residual carbon and, within the cumulative

inaccuracies of the chemical analyses, the oxygen content corresponds to that in the Sialon formulas. Therefore, the assumed reaction of SiO_2 and C is probably correct.

SUMMARY OF RESULTS AND CONCLUSIONS

The main objective of the present investigation was to produce β' -Sialons with high Si_3N_4 by pressureless sintering a mixture of separately milled β - Si_3N_4 , AlN , and SiO_2 with very small amounts of Y_2O_3 as a sintering aid. The results and conclusions drawn therefrom can be summarized as follows:

1. β' -Sialons of compositions $\text{Si}_{2.6}\text{Al}_{0.393}\text{Y}_{0.007}\text{O}_{0.4}\text{N}_{3.6}$ (Sialon A) and $\text{Si}_{2.6}\text{Al}_{0.386}\text{Y}_{0.014}\text{O}_{0.4}\text{N}_{3.6}$ (Sialon B) and densities of 3.13 and 3.17 grams per cubic centimeter, respectively, were made by pressureless sintering at 1760°C for 4 hours in stagnant nitrogen. It is concluded from these results that very small amounts of additives (or impurities) are required to effect pressureless sintering of mixtures of very fine Si_3N_4 , AlN , and SiO_2 powders.
2. The four-point modulus of rupture (MOR) of Sialons A and B range from about 460 megapascals at room temperature to 155 megapascals at 1400°C . Although Sialons with higher strengths have been reported in the literature, their compositions were either undisclosed or, if disclosed, their oxidation resistance was lower than that of the Sialons produced in this investigation.
3. The Sialons from this investigation gain weight at a rate of about $0.004\text{ mg/cm}^2\text{-hr}$ after being oxidized in air for 35 hours at 1400°C . By comparison, published results show that hot-pressed Si_3N_4 oxidizes under similar conditions, at $0.013\text{ mg/cm}^2\text{-hr}$, that a Sialon with 2.5 wt% Y_2O_3 oxidizes at $\sim 0.043\text{ mg/cm}^2\text{-hr}$, and that a Sialon containing much more aluminum (which is known to increase oxidation resistance) oxidizes at $0.0011\text{ mg/cm}^2\text{-hr}$. It is concluded that the oxidation resistance of the Sialons produced by the methods of this investigation may be increased by increasing their aluminum content.
4. After pressureless sintering Sialons A and B, their carbon content was reduced to negligible levels, and their oxygen content was lowered to the values to be expected had the carbon originally present in the starting powders reacted with SiO_2 to form CO and SiO gases. From this it is concluded that excess SiO_2 must be used on compounding β' -Sialons to compensate for losses due to reaction with carbon.

5. From the results of this investigation and a comparison with other Sialon investigations published in the literature, it is concluded that increasing amounts of Y_2O_3 increases the strength of Sialons at the expense of their oxidation resistance.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 14, 1978,
505-01.

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TABLE I. - CHARACTERIZATION OF RAW MATERIALS

Material	Source	Manufacturer designation	Purity	Size	Specific surface area, m ² /g	Chemical analysis		Spectrographic analysis or manufacturer specifications, ppm (unless otherwise noted)
						Oxygen, wt %	Carbon, wt %	
β -Si ₃ N ₄	Cerac Pure	S-1068	99.9	-325 mesh	1.50	1.70	0.019	Si major, 720 Al, 350 Ca, 30 Co, 60 Cr, 70 Cu, 60 Fe, 30 Ni, 200 W
AlN	Atlantic Equipment Engineers	AL 106	99.9	-325 mesh	1.83	2.96	0.69	Al major, 125 B, 140 Ca, 900 Co, 450 Cr, 80 Cu, 530 Fe, 40 Mg, 90 Mn, 290 Mo, <100 Na, 120 Ni, 290 Si, 190 Ti, <80 V, 340 W, <50 Zr
SiO ₂	Cerac Pure	S-1061	99.9	-325 mesh	0.92	ND ^a	0.012	Si major, 1000 Al, <10 Ca, 50 Cr, 120 Cu, 50 Fe, 190 Mg, 130 Mn, 90 Ti
Y ₂ O ₃	Moly-corp	5600	99.99	NA	8.39	ND	0.144	Y major, 130 Al, 60 Cr, 60 Cu, 123 Fe, 130 Si
HNO ₃	Eastman	13058	Reagent ACS	-----	-----	ND	ND	Residue on ignition = 3 ppm
Ethanol	IMC Chemical Group	NA ^b	200 proof	-----	-----	-----	-----	Residue after evaporation, 0.0012 wt %
n-heptane	NA	NA	NA	-----	-----	-----	-----	98.38-98.49 boiling range; specific gravity, 0.683 (20/20)
Silicone oil	Dow Corning	DC-705	NA	-----	-----	-----	-----	

^aNot determined.^bNot available.

TABLE II. - MILLING DATA AND CHARACTERIZATION OF MILLED MATERIALS

Milling data						Characterization of milled powders			
Material	Mill	Media	Fluid	Time, hr	Postmilling treatment	Specific surface, m ² /g	Chemical analysis		
							Oxygen, wt %	Carbon, wt %	Other elements, ppm (unless otherwise noted) ^a
β -Si ₃ N ₄ (S-1068)	Nickel	Nickel shot	Distilled H ₂ O	300	Leach, wash dry	10.14	2.83	0.241	0.1 wt % Al, 300 B, 130 Be, 670 Ca, 60 Cr, 180 Fe, 250 Mg, 520 Ni
AlN (Al 106)	Alumina	Alumina	n-heptane	100	Dry	8.28	4.8	0.94	<100 B, 150 Ca, 800 Co, 380 Cr, 60 Cu, 570 Fe, 60 Mn, 300 Mg, 420 Mo, 80 Ni, 630 Si, 80 Ti, <80 V, 700 W, <50 Zr
SiO ₂ (S-1061)	Nickel	Nickel shot	Distilled H ₂ O	130	Leach, wash dry	27.1	ND ^b	0.037	630 Al, 130 Ca, 70 Cr, 110 Cu, 80 Fe, 210 Mg, 900 Ni

^aValues obtained by spectrographic analysis.^bNot determined.

TABLE III. - CHARACTERIZATION OF PRESSURELESS SINTERED SIALONS

Designation	Formulas ^a	Density, g/cm ³	Percent theoretical density	Chemical analyses		
				Oxygen, wt %	Carbon, wt %	Other elements, ^{b,c} ppm (unless otherwise noted)
Sialon A	Si _{2.6} Al _{0.393} Y _{0.007} O _{0.4} N _{3.6}	3.13	^d 98.1	4.40	0.032	7.3 wt % Al, 380 Ca, 130 Co, 170 Cr, 100 Fe, 90 Mg, 410 Mo, 540 Ni, 10 Sr, 30 Ti, 90 W, 1.2 wt % Y
Sialon B	Si _{2.6} Al _{0.386} Y _{0.014} O _{0.4} N _{3.6}	3.17	^e 98.8	4.38	0.030	7.0 wt % Al, 80 B, 300 Ca, 50 Co, 130 Cr, 100 Fe, 80 Mg, 40 Mn, 210 Ni, <10 Sr, 20 Ti, 30 W, 1 wt % Y

^aSialon formulas correspond to Si_{3-x}Al_xO_xN_{4-x}.^bValues obtained by spectrographic analysis.^cEstimated accuracy, ±50%.^dBased on a density of 3.19 g/cm³ for hot-pressed Sialon A.^eBased on a density of 3.21 g/cm³ for hot-pressed Sialon B.

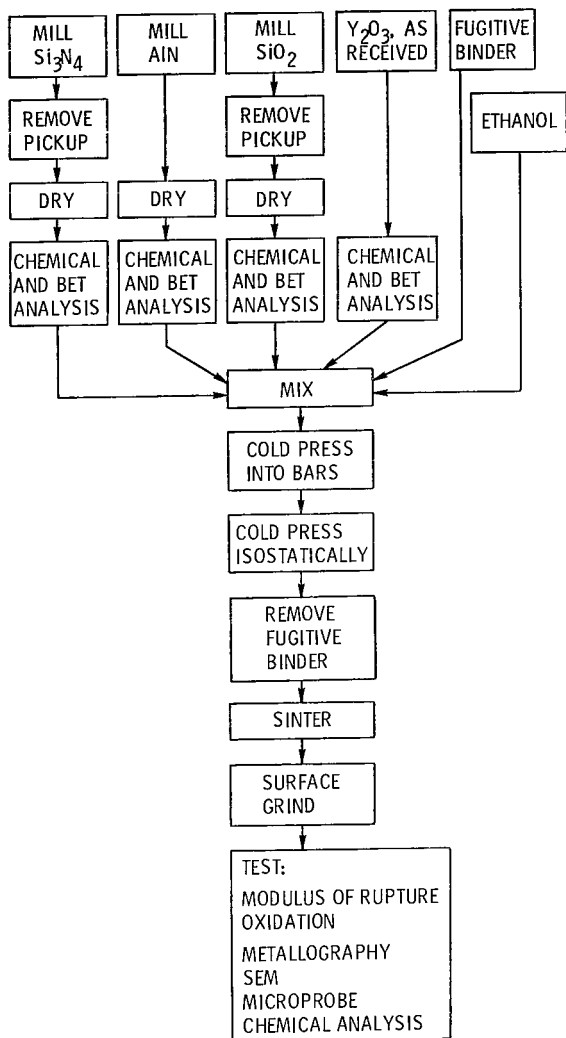


Figure 1. - Flow chart for the preparation and testing of Sialon ceramics.

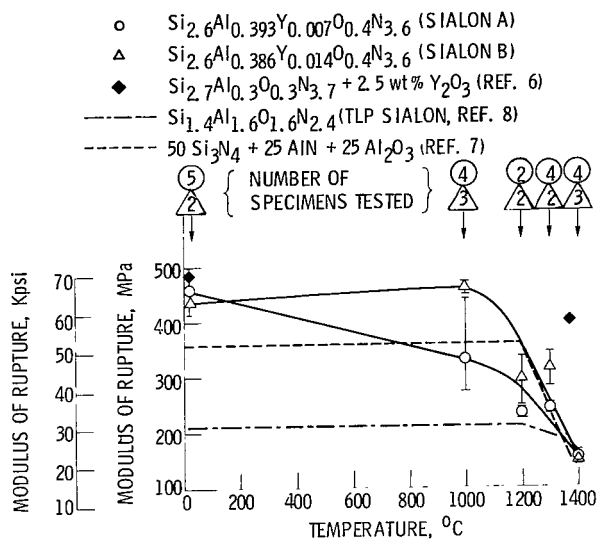


Figure 2. - Four-point modulus of rupture of various Sialons as a function of temperature.

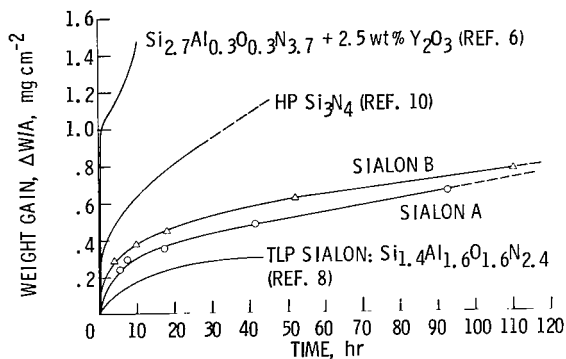


Figure 3. - Weight gain of various Si_3N_4 base ceramics oxidized at 1400°C.

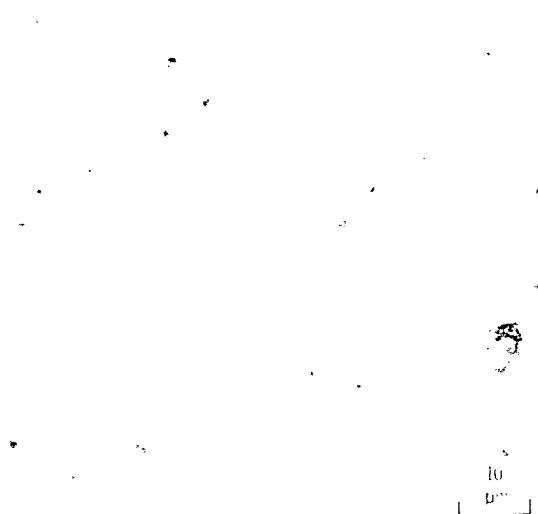


Figure 4. - Photomicrograph of Sialon A. Unetched.

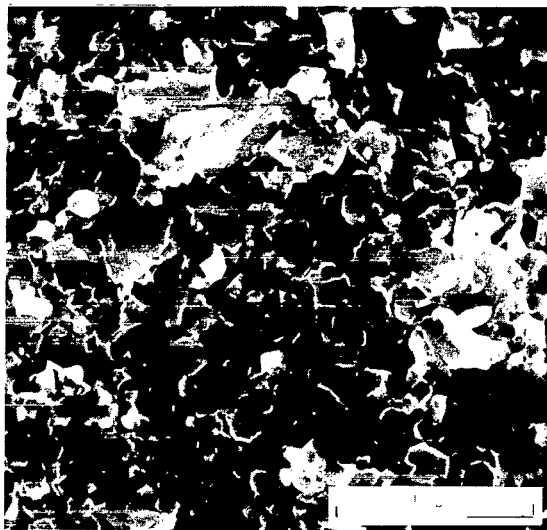


Figure 5. - Scanning electron micrograph of fracture surface of Sialon A.

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16. Abstract Two β' -Sialons of compositions $\text{Si}_{2.6}\text{Al}_{0.393}\text{Y}_{0.007}\text{O}_{0.4}\text{N}_{3.6}$ and $\text{Si}_{2.6}\text{Al}_{0.384}\text{Y}_{0.014}\text{O}_{0.4}\text{N}_{3.6}$ were pressureless sintered from mixtures of Y_2O_3 and separately milled $\beta\text{-Si}_3\text{N}_4$, AlN , and SiO_2 . These Sialons had densities of over 98% of theoretical, four-point bend strengths of 460 and 155 MPa at room temperature and 1400° C, respectively, and 1400° C oxidation rates lower than those reported for hot pressed Si_3N_4 and for a stronger Sialon with 2.5 wt % Y_2O_3 .					
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